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RESEARCH ARTICLE

Electrochemical behaviour and performance of flexible graphite filaments in different electrolytes with wide potential window of 2 V

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Abstract

Energy storage systems have received increasing attention in recent years because of the requirements of energy supply with respect to the growing population and technology. Among the technologies of energy storage devices, supercapacitors become popular due to their superior characteristics such as high power density, extremely fast charge-discharge capability and long life cycle. A wide variety of materials are already in use to fabricate supercapacitors. Carbon and its derivatives are common materials among the electrode materials of supercapacitors. In this study, electrochemical behaviour of flexible graphite filaments are investigated in different media in order to elucidate the performance of graphite as a supercapacitor material. Electrochemical experiments of graphite electrode are carried out in sodium sulphate (Na_2SO_4), hydrochloric acid (HCl), potassium hydroxide (KOH) and Ethaline deep eutectic solvent as electrolyte media. Graphite filament is cycled at wide potential window (from -1 V to 1 V) at various scan rates in the range of 5 to 100 mV s^{-1} in order to observe the associated electrochemical behaviour and performance. Graphite filament electrodes without any treatment can be used in Ethaline and aqueous Na_2SO_4 electrolytes. However, these electrodes cannot be used in acidic or alkaline media with high potential window of 2 V.

1. Introduction

The necessity of energy consumption control is popular issue for the world [1]. The primary reason for this requirement came from the fact that global energy consumption has steadily increased. Furthermore, new energy storage technologies (such as batteries and supercapacitors) have been researched commonly [2, 3]. One of the reducing the energy consumption in daily life and partly industry is to use a sustainable electric-power sources. Batteries and supercapacitors are predicted to continue to thrive as the primary utilizing mechanism in the near future. Supercapacitors can be distinguished from the batteries because of their properties such as lifespan, nontoxicity and faster charge rate. In a basic supercapacitor design contains anode and cathode electrodes [4, 5]. The electrodes are separated from each other by an aqueous or organic electrolyte. Electrolyte also facilitates ion transport across electrodes while keeping them electrically isolated from one another with a separator.

The multipurpose wearable gadgets in daily life and for military objectives can be common in the future. Smart garments consisting of supercapacitors, lithium ion batteries, solar cells could be the primary devices for wearable sensors and communication devices [6–9]. The use of wearable and portable consumable electronic devices has raised the need for supercapacitors during the last decade. With the advancement of wearable technology, there has been an increase in the demand for flexible energy storage devices that can be bent and stretched [10, 11]. Furthermore, fiber shaped supercapacitors is the new member of the supercapacitor family. This type of supercapacitors has an effective property such as light weight, small size, easy packaging, and good flexibility to be fitted on wearable electronic systems [12]. Fiber-shaped type of supercapacitors are the good candidate for energy provider of the wearable electronic systems. Fiber electrode is the crucial part of this type of supercapacitors. Electrochemical performance of the supercapacitor depends on the fiber electrode (composition, structure, stability). However, the electrochemical performance is not the only parameter for determining performance of fiber-shaped supercapacitors. Flexibility, softness and light weight may be the attractive parameters to design a fiber-shaped supercapacitors. This sort of supercapacitor may be woven into textiles without losing the fabric's characteristics softness.

General purpose of the fiber-shaped supercapacitors not only to develop the supercapacitor devices as flexible but also they should be compatible with wearable electronics, also is to keep the capacitance properties as conventional supercapacitors. Carbon electrodes and its derivatives are popular energy material for the capacitors [13–17]. Also many researchers studied the flexible supercapacitor with polymer electrodes [18–21]. Immense progresses on forming (spinning, coating etc.) and changing the electrochemical properties of different kinds of carbon based materials were successed by researchers [22]. Dry spinning [23, 24], wet spinning [25, 26] and coaxial spinning [27, 28] methods were investigated for the formation of fiber-shaped electrodes which could be used in supercapacitor applications. Different coating techniques via electrochemical deposition and dip coating method have been investigated on producing carbon based flexible supercapacitors [29–34].

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Graphite filaments could be knitted for flexible electrodes. These filaments could be coated before knitting. Alternatively, they could be knitted to obtain a flexible fabric. This fabric could also be coated by nanomaterials for electronic devices. The reason to coat a material with nanoparticles is to increase its surface area (coverage) to let more ions move to/from the surface. If more ions in an electrolyte react with an electrode, the charge could be more and higher energy could be stored. Graphite having high surface area could be coated by metals/alloys and then can be knitted for energy storage applications. In this study the usability of graphite was examined in different electrolytes. Electrochemical behavior of flexible graphite filament in Na2SO4, KOH, HCl and Ethaline electrolyte media was measured at different scan rates for each of the electrolytes by means of cyclic voltammetry tests. The electrochemical behavior of graphite filament, a carbon derivative, in various electrolytes was examined in this work without any treatment. This research was chosen as a preliminary investigation for the graphite filament based supercapacitor research. The conclusions of this investigation will be used to evaluate which electrolyte could have more acceptable electrochemical property when the graphite electrode is cycling for energy storage devices. Furthermore, it is expected that this research would lead the development of a graphite filament based supercapacitor designed by various knitting processes.

2. Materials and Methods

Pure and non-coated graphite filament samples were cut with the same length (around 3 cm). 1 M sodium sulphate (Na2SO4, Merck, 99%), 1 M potassium hydroxide (KOH, Tekkim, 90%), 1 M hydrochloric acid (HCl, Merck, 32%) and prepared ethaline (a deep eutectic solvent ionic liquid) were used as electrolyte solutions for determining capacitance properties of graphite filament in this study. Graphite filaments were cut around 3 cm length and 1 cm of them were immersed in aqueous Na2SO4, aqueous H2SO4, aqueous KOH and non-aqueous ionic liquid electrolytes to test redox reaction of graphite in these electrolytes by means of cyclic voltammogram. Cyclic voltammogram is a common electrochemical technique to reveal probably oxidation and reduction reactions of electrode or electrolyte. This technique is generally used by electrochemist to understand if a combination of electrode and electrolyte has a redox reaction. Three electrodes are generally used in this technique: working electrode, counter electrode and reference electrode. Reference and counter electrodes were mentioned in experimental part. The working electrode used in this work was graphite filament itself.

Nikon LV150NL optical microscope images were obtained to show the graphite filament structure in macro and microscale range. Measurement of capacitance behavior in different electrolytes were carried out using the potentiostat of Gamry Interface 1010 (the USA). No deposition process were applied before the electrochemical processes because only pure graphite filament was tested. Platinum coated mesh and silver with silver-chloride electrode were used as counter and reference electrode, respectively. In the electrochemical analysis, graphite thread samples were used as working electrode. There was no heat used in the study of electrochemical behavior. All specimens were scanned between the potentials of -1.0 V and 1.0 V at the room conditions (around 22 °C). Graphite filaments were cycled at different scan rates as 100 mV s^{-1}, 50 mV s^{-1}, 20 mV s^{-1}, 10 mV s^{-1}, 5 mV $s^{-1}$for all different electrolyte media. All sources of funding of the study should be disclosed. Clearly indicate grants that you have received in support of your research work.

3. Results and Discussion

Photograph and microscopic images of flexible graphite filaments are illustrated in Figure 1. Flexibility of the graphite filament is clearly shown in Figure 1a. This graphite consists of plenty of small filaments. 5x and 20x magnification of microscopic view are shown in Figure 1b and Figure 1c, respectively. It can be understood that graphite filament could have high surface area and can be used as flexible electrode and it is also easily dispersible. Therefore, it could be easily usable material for electronic devices with wearable technology.

Figure 2 illustrates the cyclic voltammogram of graphite filament in Na_2SO_4 solution. The potential window of the cyclic voltammetry started from -1 V and finished at +1 V. Indeed, the 2 V potential window is generally considered as wide potential window [35–37]. The wide potential window could be applied to graphite electrodes in order to observe all probable redox reaction. Sodium sulfate solution was prepared and graphite was polarized in this electrolyte. The length of graphite electrode immersed in that electrolyte was 1 cm. Cyclic voltammogram responses of graphite scanning between -1 and +1 V at different scan rates are presented in Figure 2. Oxidation and reduction peaks appeared when the graphite was cycled in neutral aqueous electrolyte. Current peak of the graphite scanning in neutral aqueous (Na_2SO_4) electrolyte increased upon increasing sweeping rate as expected. Charge which is directly related to energy storage can be found by integrating the area under current and time curve. As the timescale for charging (or discharging) is short at high scan rates, the current should be high to maintain appropriate charge which can normally obtain at longer timescale. Therefore, current should be high at high scan rate (shorter timescale). When the scan rate was decreased (increasing timescale), the current peak values decreased. Figure 2 illustrates that graphite could have both oxidation and reduction reactions in Na_2SO_4. Therefore, this solution could be appropriately used with graphite filament for energy storage applications.

Figure 3 illustrates the cyclic voltammogram of graphite scanned in potassium hydroxide solution by the application of different scan rates. The sweeping rate of graphite in alkaline solution was 5, 10, 20, 50 and 100 mV s^{-1}. The cyclic voltammogram responses given Figure 3 were similar upon changing scan rates. Here, Ag/AgCl reference electrode was used in KOH solution. Oxidation current between graphite electrode and alkaline electrode increased exponentially at the positive side of cyclic voltammogram. This could occur probably due to oxygen evolution reaction because no reduction reaction corresponding to oxidation reaction was observed when the potential was swept from +1.0 V to around 0 V. Additionally, reduction reaction of Figure 3 was not significantly different when the scan rate was changed. As no obvious oxidation and reduction reactions was observed when graphite filament was cycled in alkaline electrolyte, this combination (graphite electrode with KOH electrolyte) cannot be suggested for energy storage applications.

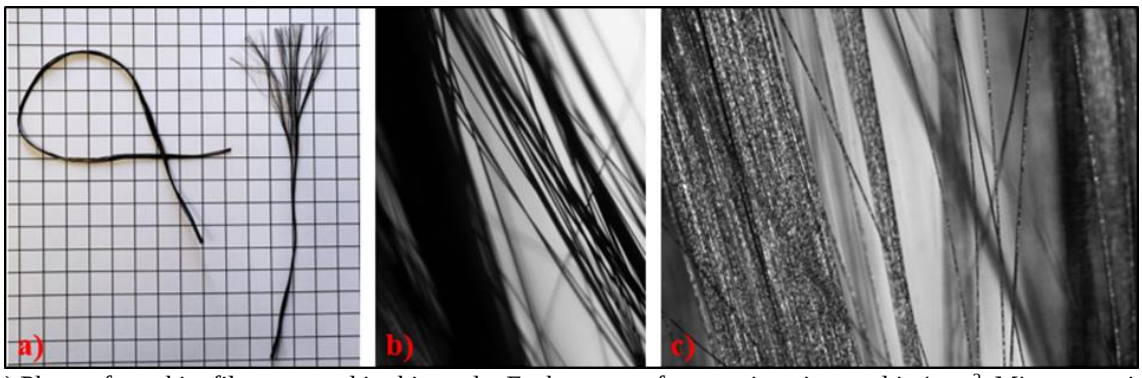

Figure 1. a) Photo of graphite filament used in this study. Each square of paper given in panel is 1 cm^2. Microscope image of graphite filament obtained by b) 50X and c) 200X magnification

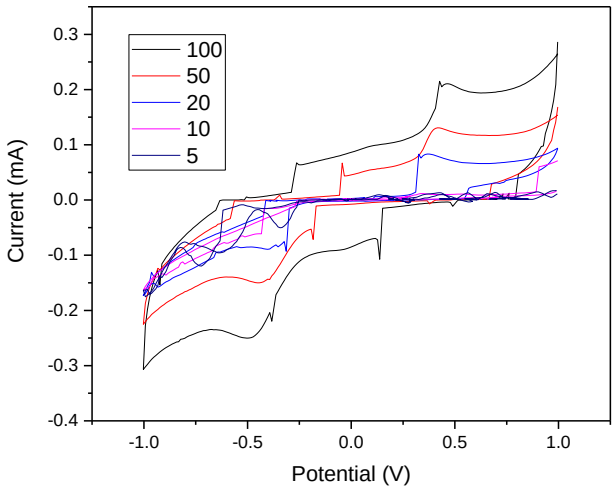

Figure 2. Cyclic voltammogram data of graphite cycling in sodium sulfate electrolyte. Ag/AgCl reference electrode was used. Numbers indicated in the graph are the scan rate of the electrode cycled in the electrolyte.

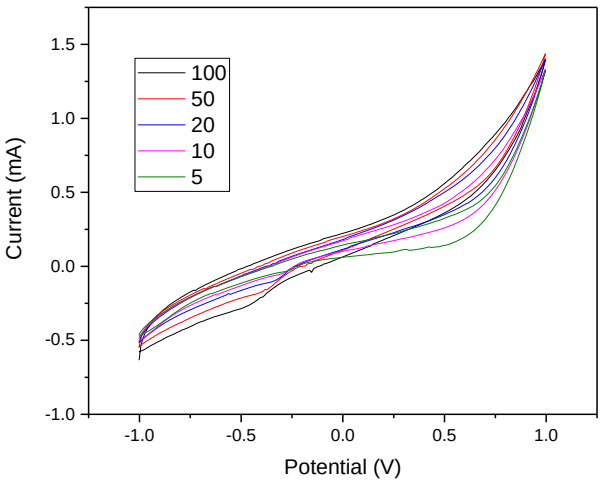

Figure 3. Cyclic voltammogram data of graphite cycling in potassium hydroxide electrolyte. Ag/AgCl reference electrode was used. Numbers indicated in the graph are the scan rate of the electrode cycled in the electrolyte.

Graphite filament was immersed in aqueous solutions having different pH (alkaline, neutral and acidic) solutions. Cyclic voltammogram responses of graphite filaments in neutral and alkaline solutions are presented in Figure 2 and Figure 3, respectively. Figure 4 shows the cyclic voltammetry of acidic solution when the working electrode was graphite filament. All graphite filaments were used directly without any further process. As the acidic solution was prepared with water, the reference electrode was again silver-silver chloride with saturated chloride solution. When the potential was increased from open circuit potential (at a potential having minimum current values) to more positive potential, current value increased dramatically (see positive side of Figure 4). Graphite was cycled in acidic solution by applying different scan rates. However, the effect of scan rate on graphite cycling in this electrolyte was not significant. The current value obtained at the scan rate of 100 mV s^{-1} (black line of Figure 4) was even less than that of 50 mV s^{-1} (red line of Figure 4). Current value at lower scan rate is normally higher than that at higher scanrate [38, 39]. As current value is not directly related to scan rate, the limiting reaction probably could not be related to electrode surface. Mass of electrolyte can be limiting reactant in this experiment. Obvious oxidation and reduction peaks are not observed when graphite was cycled in acidic solution between -1 V and 1 V (see Figure 4). Therefore, graphite filament is not an appropriate electrode in acidic medium with the conditions explained for this set-up.

After graphite filaments were cycled in aqueous solutions having different pH, a new, non-used graphite filament was transferred into non-aqueous electrolyte (Ethaline). Silver-silver chloride reference electrode is commonly used in aqueous electrolyte. However, this reference electrode could leak some its water if it is used with non-aqueous electrolyte. This leakage could affect the electrolyte conditions. Therefore, a reference electrode without water must be used in an electrolyte which does not have water. Generally, silver or platinum wire is used in non-aqueous electrolyte. In this study, a platinum quasi reference electrode was used. These non-aqueous electrolytes could be organic or ionic liquid. Graphite filament was cycled in Ethaline electrolyte and its cyclic voltammogram responses are illustrated in Figure 5.

The scan rate of the experiment was between 5 and 100 mV s^{-1} and they are shown inside the Figure 5 with different colors. The shape of cyclic voltammogram responses obtained from the interaction between graphite filament electrode and Ethaline electrolyte is similar to the cyclic voltammogram of EDLC response. Therefore, the reaction between graphite and non-aqueous electrolyte (Ethaline here) is based on a supercapacitive reaction. The current of the graphite cycling in Ethaline increased when the scan rate increased as expected. As an obvious current change was observed when graphite was cycling in Ethaline, graphite filaments could be used in Ethaline environment for energy storage applications. Graphite filaments were cycled in different electrolytes including aqueous and non-

aqueous electrolytes. Among them, neutral electrolytes (either sodium sulphate or Ethaline) could be used as appropriate electrolytes.

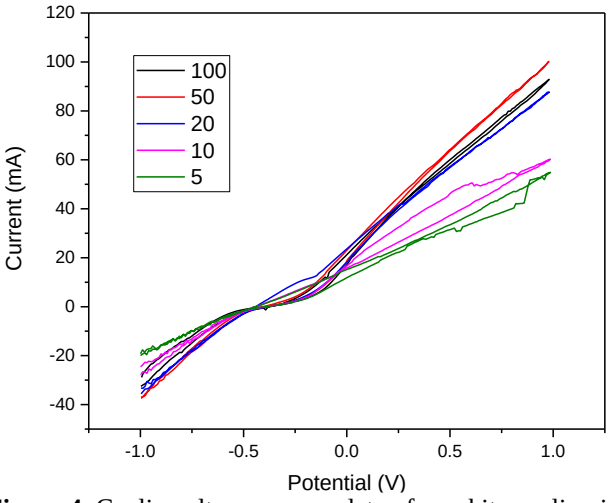

Figure 4. Cyclic voltammogram data of graphite cycling in hydrochloric acid electrolyte. Ag/AgCl reference electrode was used in ethaline. Numbers indicated in the graph are the scan rate of the electrode cycled in the electrolyte.

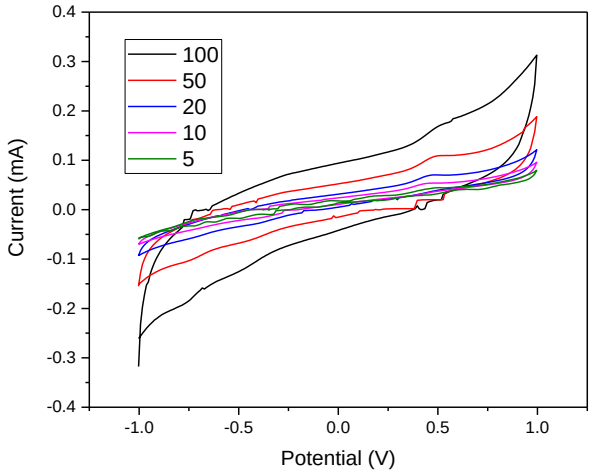

Figure 5. Cyclic voltammogram data of graphite cycling in Ethaline deep eutectic solvent electrolyte. Ag/AgCl reference electrode was used. Numbers indicated in the graph are the scan rate of the electrode cycled in the electrolyte.

4. Conclusions

Graphite can be fabricated as flexible electrode for wearable electronic applications. Graphite electrodes could be found commercially as thin sheets and they could be formed from thin filaments or compacted powders. Pure and non-coated graphite filaments could be coated with different materials and this modified electrode could have higher electrochemical performance because coated graphite could have 3D structure after coating when compare with uncoated graphite (2D). Normally filaments could be painted before knitting or weaving. Like fabrics, graphite can also be dyed after being knitted or weaving. Graphite filaments could be coated firstly before knitting or weaving. Alternatively, a fabric consisting pure graphite could be coated after it was fabricated with pure graphite. In this study, the electrochemical behavior of pure and non-coated graphite filaments is studied. This research is conducted before knitting or weaving of graphite filaments to understand the capacitance behavior of graphite filaments. Therefore, graphite filaments were immersed in different electrolytes and cycled with wide potential window. Graphite filaments were scanned in non-aqueous (Ethaline) and aqueous (having different pH) electrolytes. The aqueous electrolytes used with un-treated graphite electrodes were HCl, Na2SO4and KOH. The potential window of the electrodes scanned in different electrolytes was 2 V which is from -1 V to 1 V. Graphite electrodes consisting of filaments could be used in Ethaline and Na2SO4for energy storage applications.

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